

USP <467> Method Parameter Comparison for the Agilent 8697 and 7697A Headspace Samplers

Author

Derek W. Wolfe
Agilent Technologies, Inc.

The Agilent 8697 headspace sampler (HSS) includes significant improvements in reliability, ease of use, and instrument intelligence compared to the previous-generation 7697A instrument. A new firmware and software architecture enables fully integrated control and diagnostics for the 8697 when connected to an Agilent 8890, 8860, or Intuvo 9000 GC. These changes are designed to improve the user experience by making the instrument easier to set up, operate, and maintain.

However, many aspects of the hardware design are the same as the trusted 7697A HSS, to ensure equivalent chemical performance. The pneumatics design, thermal zones, 6-port valve, sample probe, and flow path deactivation are all carried over from the 7697A. This allows the 8697 to deliver improvements while matching the performance specifications of the 7697A.

Since the two instruments are conceptually very similar, the 8697 HSS can operate with the same method parameters as the 7697A. Headspace parameters for the USP <467> residual solvents method¹ are shown in Table 1 as an example. These parameters are used in application notes for both the 7697A^{2,3,4} and 8697⁵ headspace samplers.

Table 1. USP <467> HS method parameters for the Agilent 7697A and 8697 headspace samplers.

HS Parameter	GC/HS			
	7890/7697A	9000/7697A	8890/7697A	9000/8697
Sample Loop Volume	1 mL	1 mL	1 mL	1 mL
Oven Temperature	85 °C	85 °C	85 °C	85 °C
Loop Temperature	85 °C	85 °C	85 °C	85 °C
Transfer Line Temperature	100 °C	100 °C	100 °C	100 °C
Vial Equilibration Time	40 min	40 min	40 min	40 min
Injection Duration	0.5 min	0.5 min	0.5 min	0.5 min
Vial Size	10 mL	10 mL	10 mL	20 mL*
Vial Shaking	On, Level 2	On, Level 2	On, Level 2	On, Level 2
Vial Fill Mode	Flow to pressure	Flow to pressure	Flow to pressure	Flow to pressure
Vial Fill Flow	50 mL/min	50 mL/min	50 mL/min	50 mL/min
Vial Fill Pressure	15 psi	15 psi	15 psi	15 psi
Loop Ramp Rate	20 psi/min	20 psi/min	20 psi/min	20 psi/min
Final Loop Pressure	0 psi	0 psi	0 psi	4 psi*
Loop Equilibration Time	0.05 min	0.05 min	0.05 min	0.05 min

* The most recent application note used 20 mL vials to better accommodate a 6 mL sample volume. Also, the final loop pressure was increased to 4 psi to reduce the potential impact of atmospheric pressure variations, but 0 psi can be used without issue.

References

1. USP 32-NF 27, General Chapter USP <467> Residual Solvents, United States Pharmacopeia. Pharmacopoeia Convention Inc., Rockville, MD, USA.
2. Firor, R. L. Analysis of USP <467> Residual Solvents with Improved Repeatability Using the Agilent 7697A Headspace Sampler. *Agilent Technologies application note*, publication number 5990-7625EN, **2012**.
3. Residual Solvents Analysis Using an Agilent Intuvo 9000 GC System. *Agilent Technologies application note*, publication number 5991-9029EN, **2018**.
4. Wieder, L. *et al.* Analysis of USP Method <467> Residual Solvents on the Agilent 8890 GC System. *Agilent Technologies application note*, publication number 5994-0442EN, **2019**.
5. Eisele, I. Residual Solvents Analysis Using an Agilent Intuvo 9000 GC with 8697 Headspace Sampler. *Agilent Technologies application note*, publication number 5994-3075EN, **2021**.

www.agilent.com/chem

DE44263.6067361111

This information is subject to change without notice.

© Agilent Technologies, Inc. 2021
Printed in the USA, March 9, 2021
5994-3125EN